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PROCESS INTENSIFICATION OF LEMON GRASS OIL IN A PILOT PLANT

K. Mu'azu[1,*] A. Ganiyu[2], A. S. Alkali[3], B. Inuwa[4], A. U. Ahmed[5], S. M. Jibia[6], M. Shehu[7] and I. U. Adamu[8]

[1,2,3,5,7,8] DEPARTMENT OF PILOT PLANTS AND FABRICATION, NATIONAL RESEARCH INSTITUTE FOR CHEMICAL TECHNOLOGY, KM 4 OLD KANO ROAD, BASAWA ROAD, ZARIA, KADUNA STATE. NIGERIA
[4,6] DEPARTMENT OF INDUSTRIAL AND ENVIRONMENTAL POLLUTION, NATIONAL RESEARCH INSTITUTE FOR CHEMICAL TECHNOLOGY, KM 4 OLD KANO ROAD, BASAWA ROAD, ZARIA, KADUNA STATE. NIGERIA

E-mail addresses: [1] *kbrmuazu3@gmail.com*, [2] *emailganiyu@gmail.com*, [3] *alkaliakuso@gmail.com*, [4] *bashinuwa@gmail.com*, [5] *uzausa200@gmail.com*, [6] *muhammedengrjibia@gmail.com*, [7] *shehuyaro7@gmail.com*, [8] *uzeehauwal@gmail.com*

ABSTRACT

The extraction of essential oil from lemon-grass was carried out using steam distillation in a locally designed and fabricated pilot plant of 100 kg/day capacity. The extraction pattern and economic analysis of the oil extracted were monitored over time. The extraction revealed that 0.414 litres of oil was produced in a day of five batches with a total production cycle per batch of 1.61 hours. The study further revealed that 94.3% of the oil was extracted in 60 minutes. The GCMS result shows the major components in the extracted Lemon grass oil were Oleic acid, Neral and Citral with percentage composition of 25.69%, 19.32% and 15.38% respectively. These percent compositions compare favourably with literature values. The properties of the Lemon grass oil were found to be: specific gravity, 0.8952; iodine value, 120.7g/g; saponification value, 201.3 mgKOH/g and cetane number, 43.7. The economic analysis on a monthly basis revealed a production cost (direct and indirect) of N160,050.00K and the expected net profit of N378,150.00K.

Keywords: *Essential Oil, Lemon Grass, Extraction, Pilot Plant and Process Intensification*

1. INTRODUCTION

A new trend in our modern society is "green consumerism", which de-emphasizes overdependence on synthetic ingredients in food, flavour, and perfume. Plant-derived chemicals are "generally recognized as safe" (GRAS), and hence, they are widely being used in cosmeceuticals, food, flavor, and fragrance [1, 2, 3]. There is also increased application of phyto-chemicals in the field of medicine due to the broad spectrum of structural diversity, efficacy as phytomedicines, and utilization as intermediate compounds in drugs production [4, 5]. Essential oil which is one of the important phyto-chemicals is a mixture of various bioactive compounds [6]. Essential oil can be referred to as any concentrated, hydrophobic and lipophilic liquid of plants having high volatile aroma compounds carrying a distinctive scent, flavor, or essence of the plant [7].

This class of oils may also be called volatile oils or ethereal oils. Essential oils are found in diverse parts of plant including leaves, seeds, flowers, roots and barks. For the plant, essential oils are thought to be vital for the life of the plant, containing compounds that help to fight parasites and infections. For people, essential oils are used in perfumes, cosmetics, and bath products, for flavoring food and drink, for scenting incense and household cleaning products, and for medicinal purposes. Essential oils are specially

* Corresponding author, tel: +234 – 802 – 710 – 6787

applied in aromatherapy, which is a branch of alternative medicine where specific aromas carried by essential oils have curative effects.

Lemon-grass (*Cymbopogon Citratus*) is a rich source of lemon-scented essential oil. It is a perennial plant with long, thin leaves, and is one of the largely cultivated medicinal plants in parts of Asia, Africa and America [8]. Essential oil, extracted from the leaves of lemon-grass, possesses a wide range of biological activities like anticancer, antimicrobial, antifungal, and antioxidant properties [9-12]. The presence of these properties has made Lemon-grass oil applicable as herbicides and food preservative [13, 14]. It is also applied in the treatment of orthopedics, muscular and skin problems [10]. An important compound present in lemon-grass oil which is responsible for its bioactivities is citral. The quality of the oil is judged by the amount of citral present in the oil [15, 16].

Extraction of essential oil is conventionally carried out using solvent extraction, steam distillation, mechanical expression and hydro-distillation [17-20, 21].Among these methods, steam distillation has been the most common approach to extract the essential oils from aromatic plants in comparison to other methods such as supercritical fluid, solvent extraction, mechanical expression etc. The steam extraction method requires moderate temperature and pressure in comparison to supercritical fluid technique. In addition, the cost of solvent (water) used in steam extraction method is relatively cheaper in comparison to other solvents (hexane, methanol etc) which are commonly used in solvent extraction. The other advantage of using steam distillation is that the separation of oil from water is by gravity unlike solvent extraction where heat is needed. Extensive research work has been carried out on extraction of lemon-grass at laboratory scale using steam distillation [7, 8]. However, not much work has been done on its extraction at pilot scale. Furthermore, economics for production of lemon-grass oil at pilot plant stage is scarce in the literature. Therefore, this work aims at extraction of lemon-grass oil using steam distillation technique at pilot scale and economics of the production using liquefied petroleum gas (LPG) as energy source. This information can serve as an important resource in attracting investors to the essential oil industry and exploring its commercial utilization.

2. MATERIALS AND METHODOLOGY

The block diagram for the extraction process of essential oil from Lemon grass using steam distillation method is shown in Figure 1.

2.1 Material Sourcing and Preparation

Fresh Lemon-grass was obtained from the Division of Agricultural Colleges' plantation, Ahmadu Bello University, Zaria, Nigeria. Prior to commencing the extraction process the unwanted materials found in the Lemon grass were removed and 20kg of the Lemon-grass was weighed for a batch operation.

2.2 Extraction of Essential Oil

The extraction of essential oil was done using water as solvent in the form of steam in a steam distillation pilot plant. The pilot plant is mainly made up of oil extractor, steam boiler, condenser, gas-fired burner and oil separator as shown in Plate 1.

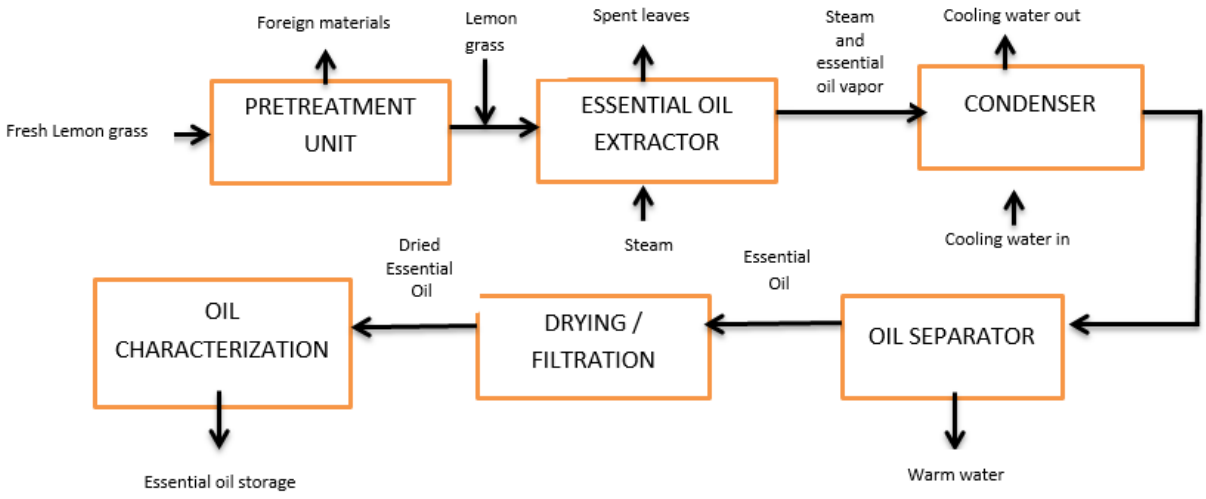

Figure 1: Block diagram for the steam extraction of essential oil from Lemon grass

KEY

A=Oil Extractor

B=Boiler

C=Condenser

D=Cooling Water Tank

E=Oil Collector

F=Steam and Oil Line

G=Cooling Water Line

Plate 1: Steam Distillation Pilot Plant for Extracting Essential Oil from Lemon Grass

The oil extractor and condenser were fabricated using grade 304 stainless steel while the oil separator was transparent glass. The boiler section is located at the bottom of the oil extraction chamber and separated by a stainless steel weir mesh. A detail of these equipment specifications is shown in Table 1.

Every component of the steam distillation pilot plant was coupled together as shown in Plate 1 for the extraction of essential oil from lemon grass [22].

The gas-fired burner is connected to a 12.5 kg gas cylinder with a rubber hose and placed directly under the oil extractor. The connections were adequately checked to avoid gas leakages. The cooling water from the overhead tank was allowed to run into the condenser at the rate of $1.67x10^{-3}m^3$/min. This was done to aid easy condensation of the steam and oil mixture vapour to liquid.

25 litres of water and 20kg (W_1) of fresh lemon grass were charged into the oil extractor. The gas line was opened and the burner was ignited and recorded the time of ignition. The burner fuel- to- air ratio was adjusted until blue flame was obtained implying steady energy supply [23]. After about 24 minutes of igniting the burner (Induction period), steam and oil mixture began to drop in the oil separator as condensate. The oil was then separated from the warm water and measured using measuring cylinder after every 10minutes for a period of 80 minutes. The oil was dried using anhydrous Sodium Sulphate (Na_2SO_4) and allowed to stand overnight followed by filtration to remove moisture and suspended impurities.

Table 1: Summary of process unit specifications

Process unit	Specification
A. EXTRACTOR	
Material of construction	Stainless steel
Height	1.65m
Diameter	0.45m
Thickness	0.003m
B. BOILER	
Capacity	$0.0075m^3$/hour
C. CONDENSER	
Type of condenser	shell and tube
Material of construction of shell	mild steel
Material of construction of tube	stainless steel
Number of passes in the tube	2
Number of pass in the shell	1
Tube length	1.2m
Tube outside diameter	0.02m
Tube inside diameter	0.016m
Number of tubes	16
Number of tubes per pass	8
Shell internal diameter	0.385m
Number of baffles	3
Baffles spacing	0.3m
Baffles cut	15%
Baffle pitch	Square
Tube side fluid	Steam and oil
Shell side fluid	Cooling water
D. OIL SEPARATOR	
Capacity	$0.0005\ m^3$
Shape	Conical
Flow rate	$0.00025m^3$/min

The same procedures were repeated for subsequent batches with addition of 10 litres of the warm water collected into the boiler as make-up water to maintain minimum of 20 litres at any time. In each case the weight of the dried oil was recorded (W_2) and oil yield was calculated using equation (1).

$$Oil\ Yield = \frac{W_2}{W_1} \times 100 \quad (1)$$

2.3 Energy Utilization

The quantity of gas (G) utilized in kg for every batch operation was calculated by taking the respective difference between the initial weight of gas cylinder (W_3) and its final weight (W_4) after the production cycle as presented in equation (2).

$$G = W_3 - W_4 \quad (2)$$

The energy utilization, U (in MJ) per batch was thereafter obtained from equation (3).

$$U = Q \times C_V \quad (3)$$

where Q is the quantity of gas consumed (kg) and CV is the calorific value of gas in MJ/kg and has the value of 46.1MJ/kg for LPG.

2.4. GC-MS Conditions

Composition of the extracted essential oil from lemongrass was analyzed by Gas Chromatography–Mass Spectrometry (GC-MS). The analysis of the essential oil was performed in the capillary column (30 m, 0.32 mm i.d., 0.25 film thickness). Column temperature was initially 40 °C for 2 minutes, and then gradually increased to 225 °C at the rate of 4 °C/ min. The extracts were diluted 3:100 (v/v) with acetone 99.99 %. Temperature of the injector and detector were set at 290 °C and 175 °C, respectively. Split ratio was set at 1:100 and the carrier gas was helium operated at a flow rate of 2.2 ml/min.

2.5. Physical and Chemical Properties of Lemon Grass Oil

The lemon grass oil was analyzed for physical and chemical properties as shown in Table 2 using standard procedures [24].

3. RESULTS AND DISCUSSION

3.1 Extraction Time

The total extraction time per batch consists of time for loading of lemon grass into the oil extractor, induction time, actual extraction time and time for off-loading the spent lemon grass as shown in Table 3.

It can be seen from Table 3 that the average time required for a batch production was 1 hour 56 minutes which comprised of actual extraction time of the oil of 80 minutes and lag period of 36 minutes. It was also observed that the average time to collect the first drop of the condensate mixture was 24 minutes (Induction period) having standard deviation of 22.3 minutes. The induction time obtained in this study was slightly higher than the value obtained in the laboratory (20 minutes) as reported in other studies [22, 25].

Table 2: Determination of physical and chemical properties using standard methods

Physical and chemical properties	Standard method	Unit
Specific gravity	ASTM D4052-91 (1995c)	-
Iodine value	AOAC CD1-25 (1993)	Centigrams l/g oil
Saponification value	AOAC CD3-25 (1997)	Mg KOH/g oil
Cetane number	ASTM D613 (1979)	-

Table 3: Total extraction time in a production cycle

Batch	Loading of fresh leaves (minutes)	Induction period (minutes)	Actual extraction time (minutes)	Off-loading of fresh leaves (minutes)
1	5	30	80	9
2	4	25	80	7
3	4	17	80	8
4	5	24	80	9
5	4	22	80	9
Average	4.4	23.8	80	8.4
Variance	0.3	4.72	0	0.89
Standard Deviation	0.55	22.3	0	0.8

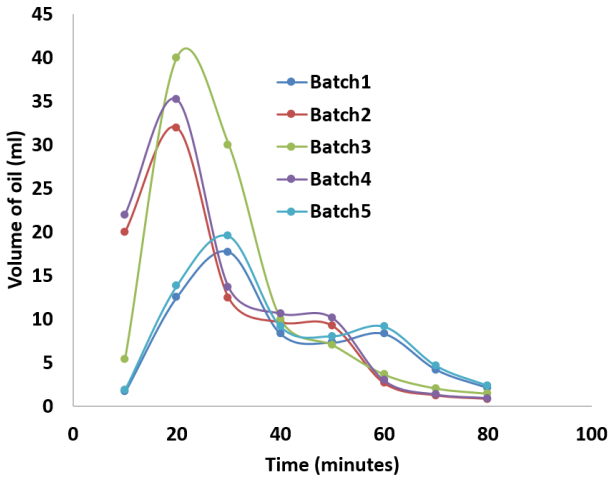

Figure 2: Extraction pattern of essential oil as a function of time

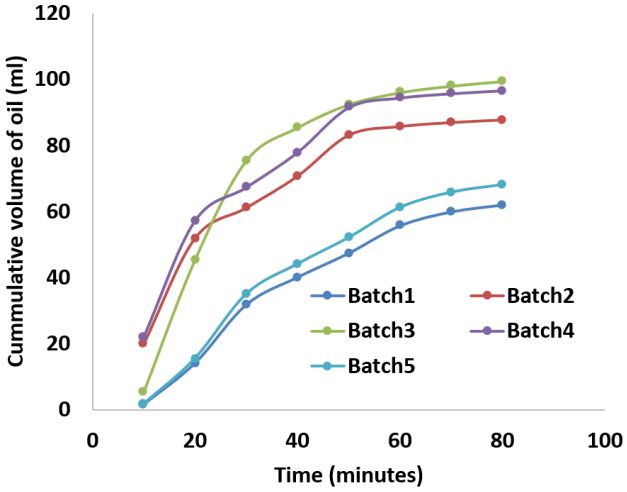

Figure 3: Cumulative volume of essential oil as a function of time

Table 4: Cumulative condensate collected in each batch

Time (minutes)	Batch 1 (ml)	Batch 2 (ml)	Batch 3 (ml)	Batch 4 (ml)	Batch 5 (ml)
10	1580	1400	1000	1100	1730
20	3260	3020	2400	2640	3580
30	5140	4660	3900	4290	5650
40	6790	6360	5200	5720	7460
50	8410	8020	6420	7060	9250
60	10130	9660	7520	8270	11140
70	12050	11260	8460	9300	13250
80	13810	12900	9380	10310	15190

However, in a separate study, the reported total extraction time of lemon grass oil in a pilot plant using steam distillation method and LPG as source of energy was 3.37 hours to obtain 87ml of oil using 22 kg of lemon grass [26]. The maximum extraction time from this pilot plant was 1 hour 56 minutes to extract 99.4ml of the oil. Based on this, the extraction time of lemon grass oil using this plant had reduced by 1.7 times (1.93: 3.37) per batch when compared to the earlier reported work [26].

3.2 Effect of Extraction Time on Oil Yield

The effect of extraction time on yield of the essential oil extracted is presented in Figure 2. It can be seen that the yield of the oil increases with increase in extraction time for all the batches until it reached 20 minutes for the 2nd, 3rd and 4th batches and 30 minutes for the 1st and 5th batches before declining. The difference in the extraction pattern between these batches could be due to loading pattern of the lemon grass. In a loosely packed lemon grass, the rate of oil extraction is faster when compared with densely packed lemon grass due to effect of pressure drop [27].

The cumulative oil extracted from the five batches is depicted in Figure 3. It can be seen that the oil extracted increases with increase in extraction time with an average cumulative volume of 82.8 ml/20kg (4.14ml/kg) per batch. This oil yield is more than 1.68 ml/kg obtained by other researchers [28].

3.3 Steam Requirement for Extraction

As seen in Table 4 the steam requirement increases with increase in the extraction time for all the five batches. However, the average steam requirement per batch was 12318 ml (12.318 litres). This implies that the steam to oil ratio was 149:1 (12318.0ml/82.8ml).

3.4 Oil Characterization

Figure 4 presents the GC-MS of the lemon grass oil extracted and detailed composition of the oil components is given in Table 5.

According to the analysis, 14 components were found in the lemon grass essential oil in which Oleic acid, Neral and Citral were the three major compounds with percentage composition of 25.69%, 19.32% and 15.38% respectively (See Table 5). In Comparison with Vietnam lemon grass oil, Myrcene, Neral and Geranial were the major three compounds found having composition of Myrcene (15.44%), Neral (29.81%) and Grenial (40.7%).The difference between the composition of these two essential oils

may be due to the differences in soil fertility and climate of the two regions [29].

The characterized properties of the extracted lemon grass oil as seen in Table 6 fall within the literature values indicating its suitability in both cosmetics and pharmaceutical industries.

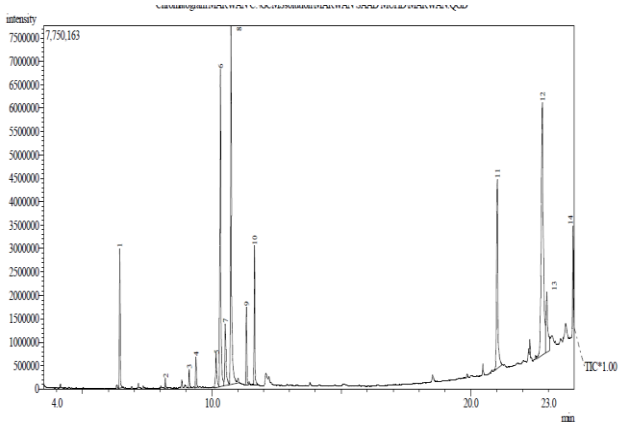

Figure 4: GC-MS analysis of lemon grass essential oil

3.5 Energy Requirement

Energy consumption plays a significant role in the extraction of essential oil from aromatic plant using steam distillation method. As seen in Table 7 the first batch consumed more energy as a result of addition of both sensible and latent heats when compared with the other batches where only latent heat was applied. It was observed that the average gas utilization per batch was 1.8 kg with corresponding energy input of 82.98MJ/kg.

3.6 Economic Analysis

Table 8 presents economics for the production of lemon grass essential oil using this pilot plant and LPG as fuel. The analysis was carried out based on the daily throughput of 0.414 litres of the essential oil and 20 working days in a month.

As seen in Table 8, the expected net profit on monthly basis based on the throughput of the pilot plant is N378,150.00. This implies that the production of lemon grass oil using this pilot plant is highly profitable and should be attractive to any potential investors. The net profit obtained in this study was higher than the net profit obtained in other study using kerosene as source of energy [29].

4. CONCLUSION AND RECOMMENDATIONS

4.1 Conclusion

The yield of lemon grass oil increases with increase in extraction time until it reached about 30 minutes before declining. Maximum yield of 99.4ml was obtained when the pilot plant was operated for 1 hour 56 minutes. The extraction time of lemon grass using this pilot plant had reduced by 1.7 times (1.93: 3.37) per batch in comparison with other plant. The physical and chemical properties of the extracted lemon grass oil were: specific gravity, 0.8952; iodine value, 120.7 cg l/g; cetane number, 43.7 and saponification value, 201.3mgKOH/g. The lemon grass oil can be used in soap, cosmetic and pharmaceutical industries.

Table 5: GC-MS results showing the components present in the oil

Peak#	Component Name	Spectrum% Area	Retention time (min)
1	Myrcene	5.05	6.435
2	β-Linalool	0.40	8.197
3	Limonene oxide	0.76	9.118
4	Cineole	1.20	9.118
5	Nerol	2.07	9.382
6	Citral	15.38	10.161
7	Nerol	3.75	10.323
8	Neral	19.32	10.512
9	Decenoic acid	2.88	10.738
10	Citronellal	5.35	11.326
11	Cineole	11.91	11.643
12	Oleic acid	25.69	21.022
13	Stearic acid	4.90	22.764
14	Berbenol	1.35	22.929

Table 7: Energy utilization for each batch operation

Batch	Quantity of gas (kg)	Energy utilized (MJ)
1	2.2	101.42
2	1.8	82.98
3	1.6	73.76
4	1.8	82.98
5	1.6	73.76

Table 6: Characterized Properties of the Extracted Lemon grass Oil

Parameter	Unit	Properties of Extracted Lemon grass Oil	Literature Values
Specific gravity	-	0.8952	0.8725-0.8965[24]
Iodine value	Centigrams l/g oil	120.7	10-185[30]
Saponification value	Mg KOH/ g oil	201.3	170-350[30]
Cetane number	-	43.7	37-51[24]

Table 8: Monthly economics for lemon grass oil production

S/N	Item	Unit	Quantity	Unit cost (N)	Cost (N)
A	**Production costs**				
1	Lemon grass	kg	2,000	20	40,000
2	Liquefied Petroleum Gas (LPG)	kg	180	320	57,600
	Water	Litre	2,000	0.5	1,000
4	Premium Motor Spirit (PMS)	Litre	10	145	1,450
5	Manpower	-	3	20,000	60,000
	Sub-total				160,050
B	**Selling price**				
1	Lemon grass oil	Litre	8.28	65,000	538,200
C	**Net Profit (A-B)**				378,150

4.2 Recommendations

(a) The effect of storage of lemon grass under sun and shade on the oil yield should be investigated using the same pilot extraction plant.

(b) The effect of parking density of lemon grass on the oil yield should be studied using the same pilot extraction plant.

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